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Original Research Article

PROXIMATE AND ULTIMATE ANALYSES OF PALM KERNEL SHELL AS PRECURSOR FOR ACTIVATED CARBON

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Abstract: This study reports the proximate and ultimate characteristics of palm kernel shell in relation to their suitability for producing highly porous carbon. The results of ad hoc samples indicated, that particle size has a decisive influence on the proximate characteristics of palm kernel shell. The effects of particle size on weight loss characteristics; rates of dehydration and de-volatilization of the carbon precursors were used to assess particle sizes that may be appropriate for carbonization. Particle sizes of 425-1180 μm are thus, suggested to be the most desirable, for the production of good quality porous carbon. This range of particles of palm kernel shell were associated with diminishing ash contents. However, the optimum particle size of the cellulosic materials that is expected to yield highly porous carbon with minimum ash contents is 1180 μm.

Keyword: Palm kernel shell, Activated Carbon, cellulosic materials.

Introduction: Carbonaceous materials that may be used as precursor for activated carbon are those with high carbon. Carbon gives the best porous absorber with excellent properties for large spectrum of industrial application such as wastewater treatment where it is used for purification, decolourization and the removal of toxic organics and heavy metal ions (Kim, *et al*, 2001).

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Activated carbon has been known as an excellent adsorbent which has been widely used due to its large adsorption capacity and low cost (Abdul Rahim *et al*, 2013). The efficacy and potential of any activated carbon depends largely on the source and the preparations. In this study, the activated carbon was produced from palm kernel shells (PKS). This agricultural waste is abundantly available in oil processing mills in Nigeria. It is an economically and environmentally sustainable raw material for adsorbents.

Characterization of the Raw Precursor (PKS)

Studies have shown that not all agricultural wastes can be used for the production of activated carbon (Billy, *et al*, 2013). There is,

therefore, the need to characterize the material for activated carbon.

The characteristics of the precursor for activated carbon are determined by proximate and ultimate analyses (Lori, et al. 2007; Barkauskas, et al, 2004; Okoroigwe and Saffron, 2012; Bilainu, 2011), to find out the compositions of the palm kernel shells as well as to determine its suitability for active carbon. Proximate analysis gives moisture content, volatile content, consisting of gases and vapor driven off during pyrolysis (when heated to 950^0C), the fixed carbon and the ash (the inorganic residue remaining after combustion in the sample). Proximate analysis is the most often used analysis for characterising coals in connection with their utilization. Ultimate analysis describes the elemental composition of biomass in wt% of carbon, hydrogen, oxygen, nitrogen and sulphur. In this study, six major characteristics have been described from which we can conveniently determine the suitability of the precursor for producing a highly porous activated carbon.

Several factors such as, the nature of the precursor as well as the amount of inorganic components, affects to a high extent, the porosity development during the process of activation (Kyotani, 2000; Yun, *et al*, 2001; Oh and Park, 2002; Zanzi, 2000, 2001). The ash content, which is the inorganic residue remaining after the organic matter has been burnt away must be very low. This is due to high ash content which indicates high inorganic constituents resulting in heterogeneous distribution of pores in the final carbon. This, however, prevents porosity evolution by blocking pore entrances during the activation process (Yun, et al, 2002). Activated carbon is generally rated on a moisture-free basis as moisture dilutes and decomposes the carbon present in the precursor hence results in very low yield of carbon (Zanzi 2000). The carbon content describes the proportion of solid carbon in the final product of carbonization. The volatile matter is the complex mixture of gaseous and liquid products resulting from the thermal decomposition of the precursor. Large carbon contents and volatile matter are, however, essential for producing highly porous materials (Lori, *et al*, 2007).

The adsorption characteristics of activated carbon from biomass such as palm kernel shells are greatly influenced by the type of carbon, which is dependent on the source of the raw materials and preparation procedures (Zanzi, 2000). However, activated carbon of different physico-chemical properties are produced from different raw material as well as their manufacturing processes, hence resulting in different adsorption characteristics (Lori, *et al.*, 2007, Lua, *et al.*, 2004). Among all the methods for water pollution control, adsorption method is considered relatively better due to convenience, easy operation and simplicity of design (Amit and Minocha, 2006).

The main objective of this study, therefore, is to investigate the proximate and ultimate characteristics of palm kernel shells relative to its suitability as precursor for highly porous carbon.

Materials and Method: Palm kernel shells were collected from a local palm kernel mill in the environs of Delta state university. The PKS was washed with tap water several times and afterwards with double distilled water four times. Then they were air-dried to prevent loss of carbon content through oven-drying. The dried PKS was crushed repeatedly to a more reduced size by a local nut cracking machine at Obiaruku, Delta State. The crushed shells were then sieved with a big rubber sieve to separate the smaller sizes from the larger sizes of the PKS. This was then taken to the Geology laboratory of Delta State University, Abraka where it was sieved into a wide range of particle sizes (2360, 1180, 600, 300, 150, and 75 µm) using a mechanical sieve, ASEW Standard Test Sieve. The sieved samples were packed into plastic containers and labelled accordingly. Proximate and ultimate analyses of pulverized kernel shells were carried out so as to guide the

selection of suitable particle size for carbonation.

The standard protocols described in official methods of analysis by AOAC (1990) were adopted for the proximate analysis of samples. Nitrogen was determined by kjeldahl method (Anderson and Ingram, 1989). Turbidimetry as described for plants by Rowell (1994) was adopted for the determination of sulphur; 0.5 g of the sample was placed in a porcelain crucible and heated in a furnace at 600^{o} C. The ash was extracted with 0.1M HCl. 5 ml of the digest was placed in volumetric flask, gelatin- $BaCl_2$ mixture was added and the absorbance at 480nm was read on UV- model 752 (UV- VIS Spectrophotometer). The amount of sulphur present was estimated by comparison of the reading with a standard curve.

To determine the total oxidative carbon, the dichromate method as described by Rowell (1994) was adopted. 1g of sample was weighed into a $250cm^3$ conical flask. 10ml of 1M $K_2Cr_2O_7$ and 20ml of $H_2SO_4$was added. The mixture was allowed to digest for 30 mins on the bench top after which 100 ml of deionized water followed by 4 drops of o-phenanthroline indicator were added. The mixture was then titrated with 0.5M ferrous ammonium sulphate [Fe $(NH4)_2$ (SO_4)] to a red end point. A blank was run in the same manner without the sample. The carbon content was determined using the formula below:

$$\text{Carbon (\%)} = \frac{B - T \times 0.003 \times 1.33 \times S \times 100}{\text{Wt of the sample}}$$

where B= amount of ferrous ammonium sulphate used in blank titration

T= amount of ferrous ammonium sulphate used in sample titration

S= strength (molarity) of ferrous ammonium sulphate

Results and Discussion: The proximate and ultimate characteristics of the palm kernel shells are shown in the table and represented with chats below. These are obviously influenced to a significant extent by the particle sizes of the precursor. The characteristics, however, vary as the sample was sieved into different particle sizes.

Fig. 1 contains the results obtained for moisture content of PKS. There was an initial fluctuation in values obtained. The moisture content ranges from 2.73 to 13.44 % with mean total moisture of 6.34% which is consistent with that obtained by Lori, *et al*, (2007) for sorghum, bagasse and millet straws. The highest and lowest moisture contents were observed for 300 and 2360 µm respectively. The moisture content obtained in this study for palm kernel shells is comparable to those reported by Okoroigwe and saffron (2012).The particle size 300 µm with a very high moisture content is likely to yield low carbon as moisture tend to decompose the carbon present in active carbon precursor (Zanzi, 2000).

The volatile content in Fig. 2 is observed to be higher with particle sizes 150 and 300 µm, and then fluctuates from 300 to 2360 µm with a mean value of 11.37%. The volatile content for PKS in this study is lower than that reported by Lori, *et al*, (2007) for sorghum, bagasse and millet straws. This may be attributed to difference in the nature of the cellulosic precursor used (Yun, *et al*, 2001; Oh and Park, 2002) which is usually characterized by different compositions of lignin, cellulose and hemicellulose. Particle sizes 300 µm though with high volatile content may not yield quality carbon of a high porosity due to the high moisture observed in Fig 1. However, particle size 1180 µm with significant volatile content may be more desirable to be used as precursor for active carbon. This deduction has been complimented by the low ash content observed (Fig. 3). The general trend in volatile matter content across the different particle sizes of PKS is expected to be replicated for the weight loss characteristics in the pyrolysis step during carbonization. From Fig. 3, it is observed that the % ash content of PKS is higher with less particle size. This is consistent with the observations by Lori, *et al*, (2007). The high percentage ash content shows large percentage

of inorganic matter, still presenting PKS with particle size 1180 µm likely to be most suitable precursor for high quality porous carbon.

The % oxidizable carbon as shown in Fig. 4 ranges from 1.86% to 3.02% with a mean total % carbon content of 2.47%. The available (inorganic) carbon needed for activation is evaluated by subtracting the organic carbon from the total carbon. The carbon contents were higher compared to those observed by Lori, *et al*, (2007) for sorghum, bagasse and millet straws. This may be due to the nature of PKS as precursor for activated carbon. Although the carbon content of PKS in the particle sizes are relatively close, the highest particle size associated with low ash content, low moisture and high volatile matter was found in particle size 1180 µm (Fig. 3). The lower carbon content observed in fine particle sizes may be as a result of the higher ash content present (Lori, *et al*, 2007; Zanzi, 2000), thereby reducing the carbon/bio-char (Okoroigwe, 2012), which is an important requirement for a highly porous carbon.

The % sulphur obtained in Fig. 5 ranges from 14,15 to 36.59% and it is lower than those reported by Lori, et al, (2007) for sorghum, bagasse and millet straws and higher than those obtained by (Bilainu, 2011). The % nitrogen obtained from particle sizes of PKS (Fig. 6) in this study ranges from 0.37% to 0.67% with a mean value of 0.53%. This is comparable with those obtained by Lori, *et al*, (2007) for sorghum, bagasse and millet straws; for PKS for bio-energy determination (Okoroigwe and Saffron, 2012) and for high ash coal and cyclone chars (Bilainu, 2011). The contents of Sulphur and Nitrogen were determined in consideration for environmental issues that may be associated with evolution of greenhouse gases that require a sink during pyrolysis of the PKS. The low values for the PKS samples suggest good characteristics as precursor for carbon adsorbents.

The values for the proximate and ultimate analyses obtained in this study reveals that the most desirable precursor for activated carbon using PKS is the particle size 1180 µm. This deduction is attributed to the relatively low ash content and high carbon content present. It is also supported by the fact that the moisture content is also relatively low.

Table 1: Ultimate characteristics of palm kernel shells

Particle size (µm)	Nitrogen (%)	carbon (%)(%)	Sulphur
2360	0.67	97.60	31.71
1180	0.59	97.07	14.15
600	0.59	96.98	21.95
300	0.47	96.35	32.68
150	0.50	95.94	28.78
75	0.37	95.73	36.59

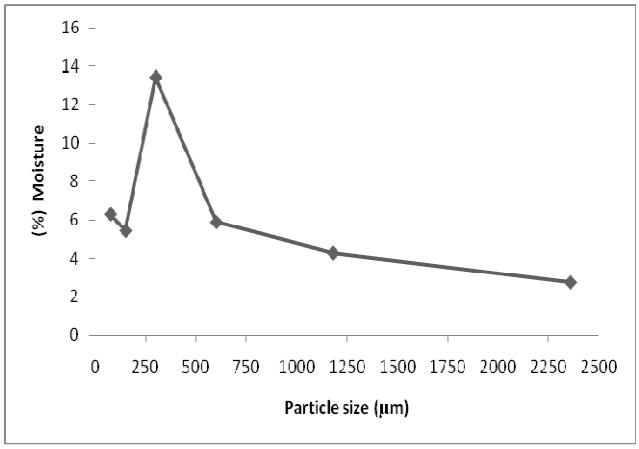

Fig 1: Effects of particle size on moisture content of palm kernel shells

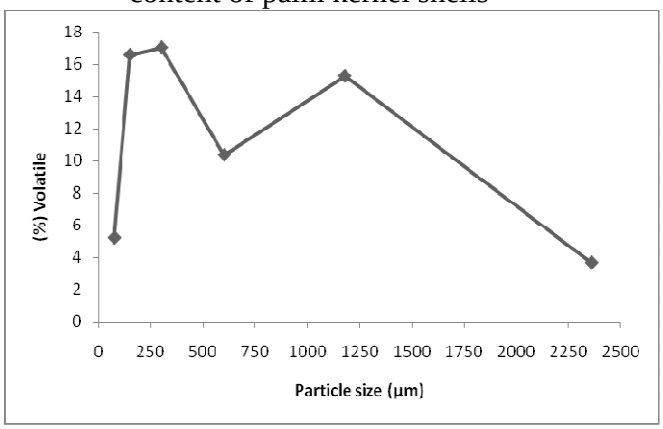

Fig 2: Effects of particle size on volatile content of palm kernel shells

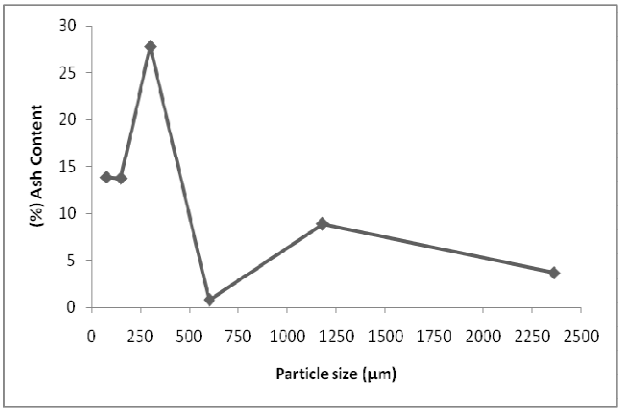

Fig 3: Effects of particle size on ash content of palm kernel shells

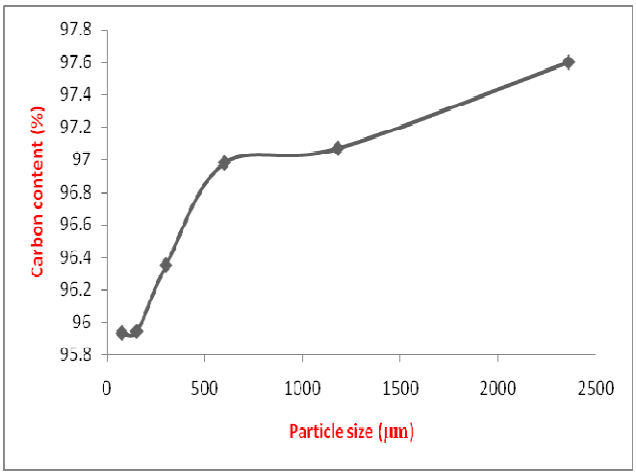

Fig 4: Effects of particle size on carbon content of palm kernel shells

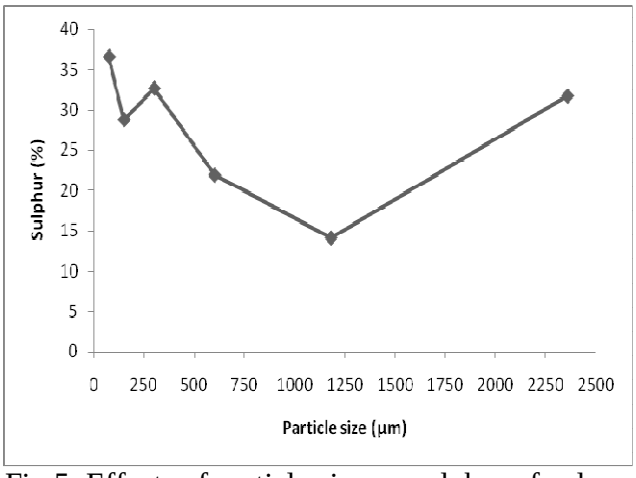

Fig 5: Effects of particle size on sulphur of palm kernel shells

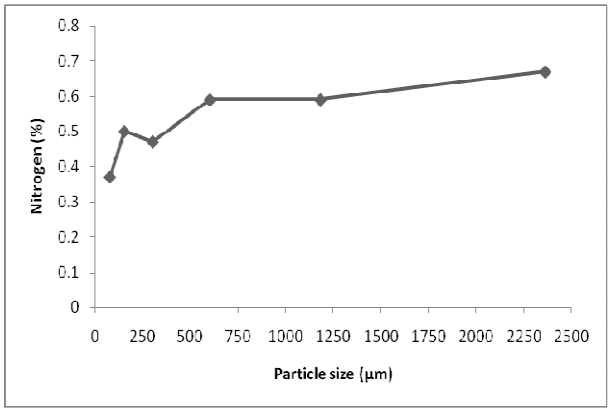

Fig 6: Effects of particle size on nitrogen of palm kernel shells

Generally, the trend of high fixed carbon associated with the larger particle sizes of PKS is peculiar to other cellulosic precursors (Lori *et al.*, *2007*). This parameter which determines the adsorbent yield is well favoured in the domain of the other proximate parameters in large particle sizes of PKS except ash content. This therefore suggests the need for solubility test of the ash in different solvents in order to determine a suitable solvent to be incorporated in the washing of the resulting carbon adsorbent following the carbonization of PKS, when large particle size of PKS is pyrolysed for active carbon.

Conclusion: From the experimental analysis carried out in this study, it is evident that chemical and elemental characteristics of palm kernel shells are greatly influenced by the particle size regarding the moisture, carbon and ash contents, which are the major determinants for a highly porous carbon. It can therefore be concluded that the optimum particle size of the cellulosic material expected to yield high quality activated carbon from PKS is 1180 µm. This affirms the useful application of PKS as precursor for active carbon in treatment of wastewater as well as drinking water, and at the same time rid our environment of agricultural wastes, such as PKS, that are causing nuisance and threat to environmental sanity.

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